

PATENT CLAIMS

1. A method for acoustic emission analysis of a granular composition comprising a biologically active compound, said method comprising colliding the granular composition with at least one surface transmitting low frequency vibrations, recording low frequency vibration data in the range of 10 Hz to less than 50 kHz, arising from the collision, with at least one vibration detecting sensor and subjecting the recorded low frequency vibration data to computerized data processing.
2. The method according to claim 1, wherein the low frequency vibrations has a frequency between 32 Hz to 25.6 kHz.
3. The method according to claim 1, wherein at least one of the low frequency vibrations is a frequency below 15 kHz.
4. The method according to claim 1, wherein the vibration detecting sensor is a piezo-electric sensor.
5. The method according to claim 1, wherein the vibration detecting sensor is an accelerometer.
6. The method according to claim 1, wherein the data processing is selected from the group of Fast Fourier Transformation, Power Spectral Density, Principal Component Analysis, Partial Least Squares Regression, Principal Component Regression, Multiple Linear Regression, Neural Network or a combination thereof.
7. The method according to anyone of claims 1-6, wherein the biologically active compound is in a purified form.

8. The method according to anyone of claims 1-6, wherein the biologically active compound is selected from bio-catalysts, therapeutic agents, herbicides, pesticides and fungicides.

5 9. The method according to claim 8, wherein the biologically active compound is selected from proteins and peptides.

10. The method according to claim 9, wherein the biologically active compound is an enzyme, particularly selected from
10 hydrolases and oxidoreductases.

11. The method according to claim 1, wherein the granular composition further comprises auxiliary granulation agents.

15 12. The method according to claim 11, wherein the auxiliary granulation agents are selected from fibre materials, binders, fillers, liquid agents, enzyme stabilizers, suspension agents, cross linking agents, mediators and/or solvents

20 13. The method according to claim 1, wherein the granules comprises a core wherein the biologically active compound is intimately mixed with auxiliary granulation agents.

14. The method according to claim 1, wherein the granules
25 comprise a core particle coated with a layer comprising the biologically active compound and preferably auxiliary granulation agents.

15. The method according to claim 1, wherein the granules have
30 an average size between 20-2000 μm , preferably between 100-1000 μm , more preferably between 200-800 μm .

16. The method according to claim 1, wherein the granules are coated with a coating agent.

17. A process for preparing granules comprising a biologically
5 active compound and optionally auxiliary granulation agents in a granulation apparatus said process comprising the step of performing acoustic emission analysis on the granules in accordance with claims 1-16 on the granules forming in the granulator.

10 18. The process of claim 17, wherein the acoustic emission analysis is performed on-line and in real time during the granulation process and is repeated more than one time during the granulation process.

15 19. The process of claim 17, further comprising the step of changing at least one process parameter as a result of the acoustic emission analysis.

20 20. The process of claim 19, wherein the process parameter is selected from supply of biologically active compound, supply of auxiliary granulation agent, supply of coating agent, supply of gas, temperature, pressure, pH, speed and mechanical force conferred to the process.

25 21. The process of claim 17, further characterized by being a coating process wherein granules comprising a biologically active compound and optionally auxiliary granulation agents are coated with a coating agent and the parameter is supply of
30 coating agent to the granulation apparatus.

22. A granulation or coating apparatus comprising

(a) a granulation or coating device comprising at least one chamber for processing material into granules or coated granules,

5 (b) an arrangement for performing acoustic emission analysis comprising at least one vibration detecting sensor capable of detecting vibrations in the range of 10 Hz to less than 50 kHz and optionally amplifying and filtering units and a computer unit.

10 23. The apparatus of claim 22, wherein the granulating or coating device is selected from a fluid bed granulator or coater, high shear mixer granulator, a coating mixer, a spray dryer, a spray cooler, an extruder.

15 24. The apparatus of claim 23, further comprising means for providing a purge stream of granules from the chamber and wherein the optical arrangement is positioned to allow fluorescence analysis of granules in the purge stream.

20 25. The apparatus of claim 23, further comprising one or more elements selected from computing units and control units

26. Use of acoustic emission analysis in the process of making granules comprising a biologically active compound.